

Patent Application No. 09/880,737

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Denes *et al.*

Serial No.: 09/880,737

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Docket No.: 032026-0538

Examiner: K. Mayekar

For: **METHOD AND APPARATUS FOR PRODUCING COLLOIDAL
NANOPARTICLES IN A DENSE MEDIUM PLASMA**

DECLARATION OF FERENCZ S. DENES

I, Ferencz S. Denes, do hereby declare and state as follows:

I have been involved in the field of plasma chemistry for more than 17 years. I have been employed at the University of Wisconsin for the last 14 years. I presently hold the position of associate professor.

I received a Master of Science in Chemical Engineering from Gh. Asachi Polytechnical Institute, Jassy, Romania in 1965 and a Ph.D. in Chemical Engineering from Gh. Asachi Polytechnical Institute, Jassy, Romania in 1972.

My curriculum vitae is attached hereto as exhibit A.

I am an inventor of the subject matter claimed in U.S. Patent Application Serial Number 09/880,737, filed on June 13, 2001. As amended, independent claims 1, 44, and 52 in the above-referenced U.S. Patent Application reads as follows:

1. A method for producing a colloidal dispersion of nanoparticles in a dense fluid medium, the method comprising the steps of:
 - (a) providing a reaction vessel for containing the dense fluid medium;
 - (b) charging the dense fluid medium into the reaction vessel;
 - (c) providing a rotatable first electrode comprising a first conductive material and terminated in a ceramic pin-array holder, the rotatable first electrode having (i) an end piece with a planar surface and (ii) an array of pins mounted in the pin-array holder and the planar surface; the first electrode immersed within the dense fluid medium;
 - (d) providing a static second electrode comprising a second conductive material and having a planar surface, the second electrode immersed within the dense fluid medium and being near to the first electrode;

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- (e) rotating the first electrode such that the dense medium is circulated between the first and second electrodes; and
- (f) imposing an electric potential between the rotating first electrode and the second electrode to create electrical discharges between the pins and the planar surface of the second electrode, the electric potential being sufficiently high to dislocate nanoparticles (as a result of the arclets formation) of at least one of the first conductive material or the second conductive material from the respective electrode.

44. A method of producing colloidal nanoparticles comprising:

- (a) providing a dense medium plasma discharge apparatus comprising:
 - (1) a chamber forming a reaction vessel for a dense medium;
 - (2) a first electrode mounted for rotation about an axis in the chamber having an end piece of conductive material with a planar surface, the first electrode terminated in a ceramic pin-array holder;
 - (3) a plurality of pins in an array projecting from the planar surface and mounted in the ceramic pin-array holder and the planar surface of the first electrode;
 - (4) a second electrode mounted in the chamber and having an end piece of conductive material with a planar surface;
- the planar surfaces of the end pieces of the first and second electrodes separated from each other by a gap;
- (b) immersing the first and second electrodes in a dense medium;
 - (c) rotating the first electrode with respect to the second electrode; and
 - (d) imposing an electrical potential between the first electrode and the second electrode to create electrical discharges between the pins and the planar surface of the second electrode to form nanoparticles between the first and second electrodes.

52. A method for producing a colloidal dispersion of nanoparticles in a dense fluid medium, the method comprising the steps of:

- (a) providing a reaction vessel for containing the dense fluid medium;
- (b) charging the dense fluid medium into the reaction vessel;
- (c) providing a rotatable first electrode comprising a first conductive material and terminated in a ceramic pin holder, the rotatable first electrode having (i) an end piece with a planar surface and (ii) at least one pin made of the first conducting material mounted in the ceramic pin holder and the planar surface of the first electrode; the first electrode immersed within the dense fluid medium;
- (d) providing a static second electrode comprising a second conductive material and having a planar surface, the second electrode immersed within the dense

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fluid medium and being near to the first electrode, wherein the planar surface of the first electrode is substantially parallel to the planar surface of the second electrode;

(e) rotating the first electrode such that the dense medium is circulated between the first and second electrodes; and

(f) imposing an electric potential between the rotating first electrode and the second electrode to create electrical discharges between the pins and the planar surface of the second electrode, the electric potential being sufficiently high to dislocate nanoparticles of at least one of the first conductive material or the second conductive material from the respective electrode.

The ceramic pin (array) holder in the methods recited in independent claims 1, 44 and 52 acts as an insulator, preventing electrolysis from developing in the dense fluid medium between the planar surface of the end piece of the first electrode and the planar surface of the second electrode, while permitting electrical discharges between the pins and the planar surface of the second electrode. If the ceramic pin (array) holder is not present, electrolysis will develop in the dense fluid medium and the electrical discharges necessary to produce the nanoparticles will be extinguished.

This electrode configuration permits the discharge to run in both dielectric and conductive liquid media (e.g. benzene, water, etc.) and allows the generation of nanoparticle systems both from the electrodes and the liquid media (hybrid nanoparticles) or especially from the electrodes (e.g. generation of silver nanoparticles in water).

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: April 20, 2006

Signature: 
Ferencz S. Denes